AD-A159 949 OPTICAL SURFACE ROUGHNESS AND SELECTIVE ETCHING OF INTERDENDRITIC REXILLIUM III PHASES(S)(U) NAVAL DENTAL RESEARCH INST GREAT LAKES IL J R KELLY ET AL AUG 85 1/1

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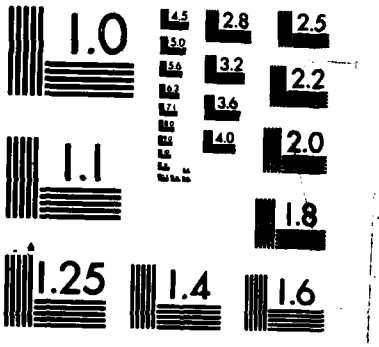

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J. R. KELLY
V. P. THOMPSON

NAVAL
DENTAL RESEARCH
INSTITUTE

Naval Medical Research and Development Command
Bethesda, Maryland

NAVAL DENTAL RESEARCH INSTITUTE
NAVAL TRAINING CENTER, BUILDING 1-H
GREAT LAKES, ILLINOIS 60088-5259

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V. P. THOMPSON

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Approved and released by

G. E. CLARK
Captain, Dental Corps
United States Navy
Commanding Officer

INTRODUCTION

Eletrolytic etching involves the differential removal of metallic phases or compounds in order to develop a mechanically retentive surface. The success of this technique depends upon, 1) factors which influence the cast metal microstructure and 2) variables affecting the selective removal of one phase(s) versus another. Laboratory etching variables have traditionally been studied by measuring resin tensile bond strengths and by scanning electron (SEM) and light microscopic visualization of etched surface architecture.

Tensile bond testing is obviously important for the final documentation of adhesion characteristics, comparing different alloys and for studying the clinical manipulation of well etched metal. Bond strength testing, however, cannot provide much detailed information about the initial stages of etching or stages of electropolishing. Both of these are of interest in obtaining an overall understanding of the etching behavior of a particular alloy. Bond strength results are dependent upon many mechnical testing variables, can vary with metal surface wettability and the bonding products used, and require the testing of a large number of samples for statistical significance between independent variables. Microscopic visualization can dramatically document the surface conditions but is laborious to quantitate and therefore usually reserved for descriptive analysis.

This work further investigates the use of biangular reflection photometry as a quantitative method for studying the development of retentive metal architecture. Reflection photometry has been shown to register very early surface area changes occurring on the etched metal (1). This phenomenon was used to study a number of electrolytic etching variables over ranges, and with a precision, not obtainable by tensile bond studies. Further, this study investigated the relationship between bond strength data and photometric data for a number of different dental laboratory etching variables. Various optical characteristics of the experimental photometer were also investigated to improve upon its design for this purpose.

BACKGROUND

A biangular measuring technique was chosen over total hemispherical reflection for two basic reasons. First, specular reflectance measurements are reported to steadily decrease with increasing metal surface roughness, whereas total hemispherical values become virtually independent of roughness beyond a certain surface roughness (2). Secondly, biangular measurements require less sophisticated and more readily available equipment than do hemispherical techniques.

Fiber optics were chosen as the basis of the photometer for reasons of availability, simplicity and later ease of designing

features of the laboratory instrument into a device for quality control use in dental laboratories. The narrow acceptance angle of fiber optics reduces noise contributions from ambient light and provides sufficient control over incident and viewing solid cone angles. Stable positioning devices, light sources, filters and photodetectors are available as integrated systems covering a very wide range of size, sophistication and cost.

Effects of surface roughness on biangular reflection have been studied for many materials, including nickel, aluminum and roughened glass (3,4). Statistical models have been successfully developed for the mathematical description of roughened surfaces so that an optical root mean square surface roughness can be calculated from reflection data (5). However, these models are generally applicable only for situations where 1) the surface details do not appreciably shadow each other (5) and 2) the surface details are much smaller than the wavelengths of radiation used (6). For visible light, 10% errors in reflection measurements will occur for a root mean square surface roughness of approximately 150 angstroms (6) and such errors increase as a logarithmic function of surface roughness. Mathematical models of visible light reflection from extremely rough surfaces do exist (7,8) but are quite complex, require a regular distribution of the modeled features and seem to provide little for direct application to the study of etched dental alloys.

Instrument variables important in the measurement of reflected light include 1) angles of incidence and viewing, 2) viewing aperture, 3) polarization effects, 4) wavelength effects as well as 5) the root mean square surface roughness (3,9,10). It was assumed that many of these same variables would affect the reflection of light from extremely rough surfaces as well and were studied as part of this investigation.

Dental laboratory variables studied by the light reflection method include 1) etch time, 2) sulfuric acid concentrations, 3) methanol concentrations, 4) etch bath temperature, 5) aging of the etch bath, and 6) current densities. Many of these conditions were found to be inter-related and were studied as covariables. Bond strength testing was performed on five sets of reflection-characterized discs, representing the independent variables of etch time, current density and temperature. Light and SEM photomicrographs were obtained to provide descriptive evidence of the surface changes quantitated by both techniques.

MATERIALS AND METHODS

The reflection photometer, as photographed in Figure 1, consisted of a tungsten light source (Fiber-Lite model 170-D, Dolan Jenner), fiber optic cables (BT624 Dolan Jenner), a silicon photodiode irradiance probe (J6512, Tektronix), and a digital photometer (J16, Tektronix). The light source was powered via a variable voltage transformer (Staco, Inc.) and power to this and

the digital photometer was fed through a voltage surge suppressor (No. 6676 Inmac). Both the viewing and irradiating optical cables were attached to vertically positioned 360-degree rotating stages (Ealing Optics) having a vernier scale for reading angular position to 0.5 degrees. These positioning devices were clamped to steel posts allowing for height adjustments and rotation within the plane of the table top. The steel posts were positionable in an array of locations via threaded fittings in a standard optical table (Oriel Corporation). Angles of incidence and viewing were measured from the surface normal. Cable alignment was always maintained at 180 degrees in the sample plane.

Study samples were cast of Rexillium III (Rx Generic) using small plastic disc patterns (r=8.0 mm, t=1.0 mm). These samples fit into a shallow (0.5 mm) well, positioned over the center of a small rotary stage (Ealing Optics) capable of 360 degrees of rotation in the plane of the optics table. A coating of flat-black paint covered the sample stand. This stand was calibrated to 1.0 degrees. Reflection measurements were generally recorded from discs at 0, 60, 120, 180, 240, and 300 degrees of sample rotation in the table's plane. These six measurements would often differ slightly due to the numerous grain colonies exposed by etching having differing crystallographic orientations accentuated by the anisotropic nature of electrolytic etching. These six readings were averaged.

Visible light interference filters (Oriel Corporation) were fit into the irradiance probe between its silicon diode and the terminal end of the viewing fiber optic cable. These filters had a bandwidth of 70 nm at 0.5 peak height. Linearly polarizing lenses (Oriel Corporation) were mounted over the irradiating and viewing optical cables proximal to the sample being studied. The viewing area of the viewing fiber optic cable was determined by briefly connecting it to the light source. When necessary the viewing-cable-to-sample distance could be adjusted with calipers (i.e., during changes in viewing angle). An additional technique for resetting the viewing optical cable made use of a third optical cable, which rested on the sample stage and accepted light at a right angle to its long axis. In this way height adjustments could be made to the viewing cable by irradiating through it, measuring with the third cable and adjusting the sample table height until the proper light intensity was obtained. Consistent irradiation of the sample was similarly maintained during experiments requiring sample table height adjustments. Control measurements were taken from the flat-black sample holder, sans sample, at the 0 degree setting of the rotating stage.

Discs were induction cast (Ticonium Co.) following burn-out at 1750 F. Samples were smoothed to a flat surface with rotary instruments followed by polishing with 240 grit carborundum paper on a metallurgical sample surfacer. The surface to be etched was air-abraded with 50 micron aluminum oxide. Electrolytic etching (EPC-100, Advanced Dental Sciences) was carried out in 200 ml of the indicated solution at the indicated current density and time

for each experiment. Both time and current densities were digitally controlled with the ADS machine. Etched discs were separated from their electrodes and mechanically cleaned of wax and electrode paint. A slotted plastic cylinder capable of holding six discs was used to hold the discs for cleaning. Discs were ultrasonically cleaned in 18% HCl for 10-12 minutes, rinsed in deionized water and then ultrasonically cleaned in 200 ml deionized water for 60 seconds.

Etch bath temperatures, where indicated, were maintained by placing the etching beaker in either a water, water-ice or water-ice-methanol bath. The etch solution temperature was directly monitored with a mercury thermometer. Where etch bath temperatures are not indicated, etching was performed at the prevailing room temperature. All etch solutions and cleaning solutions were prepared with deionized water and reagent grade acids or solvents. Etched discs were viewed and photographed by dark field light micrscopy (Nikon Epiphot). Representative discs were viewed and photographed by SEM (JSM-35, JOEL Ltd.).

Alloy discs were rinsed in acetone and air dried for ten seconds. Each disc was positioned in a bond alignment apparatus (11) and a thin layer of freshly mixed bonding agent (Enamel Bonding Agent, L.D. Caulk) applied. Immediately, a stainless steel tube (6 mm O.D. x 2 mm I.D.) filled with composite resin (Comspan, L.D. Caulk) was aligned and centered on the etched alloy surface. This is a larger diameter tube and lacked a bevel, as compared to the previous work (11). Samples were removed ten minutes after bonding, placed into 37 C water and thermally cycled for a minimum of 1000 cycles from 5-50 C prior to bond testing. The tensile bond test employed has been previously described (11). The strain rate was 1 mm/min. in all cases. Samples were routinely inspected using the stereomicroscope (60X) to determine the bond failure mode.

Statistical analysis was performed on a microcomputer using commercially available software. Means and standard deviations were calculated from the six reflection readings (0, 60, 120, 180, 240 and 300 degrees) for each disc. These single disc means were used to calculate means and standard deviations for sets of 3-10 discs representing different etch or optical conditions. Curve fitting was performed to test for mathematical relationships between etch variables.

DENTAL LABORATORY ETCH VARIABLE RESULTS

1. Sulfuric acid concentration

Eight different concentrations of acid were tested; 1, 2.5, 5, 10, 15, 16, 18, and 20 percent. Current density, etch time and etch bath volume were held constant at 300 ma/cm^2, 180 secs and 200 ml. Mean reflection values are graphically presented in Figure 2 (angle of incidence (AI) = 45 degrees, angle of viewing (AV) = 50 degrees).

2. Current Density (at room temperature)

Five separate current density settings were employed; 100, 200, 300, 400, and 500 ma/cm^2. Acid concentration and etch time were held constant at 10% and 180 seconds. Mean reflection values (AI=45, AV=50) are plotted in Figure 3. Tensile bond data is listed in Table 1.

3. Aging of Etch Solution

Two discs (4 cm^2 total surface area) were mounted on one electrode and etched for up to 30 times (300 ma/cm^2, 200 ml, 10% sulfuric acid, 180 seconds). This "test bridge" was air-abraded to expose unetched metal following each etch period. Discs used for reflection measurements were etched in the same solution, following etching of the "test bridge" for 0, 5, 10, 15, 20, 25, and 30 times. Reflection data (AI=45, AV=50) are presented in Table 2. No effect was seen for up to 30 "etched bridges".

4. Methanol Concentration

Methanol was added to the 10% sulfuric acid bath to create solutions containing the alcohol in 0, 5, 10, 15, and 20 volumes percent. Current density and etch times were held constant at 300 ma/cm^2 and 180 seconds. Reflection data are presented in Table 3 (AI=45, AV=50). Changes in methanol concentration did not seem to affect surface roughness.

5. Etch Bath Temperature

The effect of etch temperature was investigated between 5 and 45 C. Two conditions of current density and etch time were studied; 1. 200 ma/cm^2 for 420 seconds and 2. 150 ma/cm^2 for 180 seconds. Reflection data from these experiments are in Figures 4 and 5 (AI=45, AV=50). Tensile bond strength means versus etch bath temperature are presented in Figure 6.

6. Etch Times

Etch times were varied from 60 to 660 seconds at 200 ma/cm^2 at 25 C. Reflection means (AI=45, AV=50) are plotted in Figure 7 and bond strength means in Figure 8.

7. Etch Time and Current Density (5 C)

Etch times were varied from 30 to 420 seconds with the acid bath maintained at 5 C. Three conditions of current density were evaluated; 100 ma/cm^2, 200 ma/cm^2, and 300 ma/cm^2. Reflection data (AI=45, AV=50) from these experiments are plotted in Figure 9.

PHOTOMETER OPTICS RESULTS

Optical experiments were performed to better understand and to enhance the use of fiber optic, biangular reflection for measuring retentive surface roughness on dental alloys.

1. Effect of Viewing Angle (AI=45, white light)

Viewing angles were varied between 20 and 60 degrees, relative to the surface normal. Unfiltered light was used, incident at 45 degrees as in the laboratory variable experiments above. Mean reflection values are presented graphically in Figure 10.

2. Effect of Viewing Angle (AI=30, white light)

This experiment was run as above, but at a smaller angle of incidence. Reflection data are presented in Table 4.

3. Effect of Viewing Angle (AI=30, 702.5 nm)

The shift of the reflection peak away from the angle of incidence, as seen in Figure 10 and Table 4, led to the speculation that a diffraction effect was being seen. To investigate this further, experiment number 2 above was repeated using filtered light. Data from this experiment are presented graphically in Figure 11. Control readings were taken from the flat-black specimen stage.

4. Linear Polarization Effect

Polarizing filters were secured over the irradiating and viewing ends of the fiber optic cables. These filters were positioned in four separate orientations:

1. incident and viewing E vectors parallel, both perpendicular to plane of incidence;

2. incident and viewing E vectors parallel, both parallel to the plane of incidence;

3. incident vector perpendicular to the plane of incidence, viewing vector parallel;

4. incident parallel with and viewing vector perpendicular to the plane of incidence.

Control readings were taken at the same time with both polarizing filters removed. Reflection values from these trials are plotted in Figure 12.

5. Viewing Aperture Effect

Two separate fiber optic cables were used for viewing the reflected light. The first had a diameter of 1 mm and the second a diameter of 3 mm. Both cables had the same manufacturer, transmission characteristics and length. Both viewed approximately the same disc surface area, as judged by first irradiating through the viewing cable. The discs used for this work were etched for 60 to 180 seconds at 300 ma/cm^2. A ln-ln plot of reflection values vs. time is linear for this portion of the etch curve (1). Such a ln-ln plot for the aperture size data is presented in Figure 13.

REFLECTION-BOND STRENGTH CORRELATION RESULTS

Five sets of discs representing different etch conditions were jointly studied by reflection and tensile bond strength tests. These experiments included etch bath temperature (two sets), current density (one set) and etch time (two sets). Three to five discs were used per condition.

Reflection and bond strength correlated very well for one etch temperature set ($r=.94$, $F=24.82$, $df=1,4$, $p<.01$) and one etch time set ($r=.96$, $F=22.67$, $df=1,3$, $p<.05$). The linear regression plot for the temperature set is Figure 14.

Both the reflection and bond strength curves were similar for the other three experiments, in that they both generally predicted the same minimum and maximum bond strength conditions. However, no statistical correlation was found; current density experiment ($r=.63$, $F=2.0$, $df=1,4$), etch temperature experiment ($r=.62$, $F=1.25$, $df=1,3$) and etch time experiment ($r=.70$, $F=2.02$, $df=1,3$).

This lack of linear correlation significance may well have resulted from a combination of (1) the large coefficient of variation in bond strength measurements, (2) the use of too few independent variable conditions and (3) resulting limitations on the degrees of freedom. Interestingly, all the bond data display a very good parabolic fit to the independent variables etch time and temperature (Figures 6 and 8) as is the case for the reflection data (Figures 5, 7 and 9). When a few more "data points" are generated using the appropriate polynomial expressions, the bond strength and reflection data are well "correlated" for all the experiments. Under these conditions, however, statistical significance cannot be properly measured by methods of linear regression.

DISCUSSION

The most general finding of this study is that surface roughness, as measured by reflection, may be represented by a second order polynomial in two of the independent variables

employed, etch time and temperature (Figures 5, 7 and 9). Tensile resin bond strengths were found to behave in this same manner (Figures 6 and 8). This was not true for sulfuric acid or methanol concentrations or for the aging of the etch solution. The same optimum current density (200 ma/cm^2 at room temperature) was predicted by both reflection and bond strength measurements (Figure 3 and Table 1).

An interesting specific finding was the effect of temperature on the development and nature of the etched surface. As can be seen in Figures 4, 5, 6 and 9 the interplay between temperature and current density is profound and not necessarily expected. At 5 C Rexillium III is quickly electropolished at 200 ma/cm^2. At lower temperatures the current density must be reduced in order for selective etching to occur. Conversely, it was found that at higher temperatures only an annular area on the disc's periphery was well etched. This may indicate that a higher current density is needed to produce selective etching as the etch bath temperature is raised. The SEM photomicrographs in Figure 15 dramatically demonstrate the difference in selectivity towards the interdendritic phase(s) at 5 C and 35 C for a current density of 150 ma/cm^2.

There seems to be no general agreement on a basic theory to explain all the measurable aspects of pitting corrosion (12). Empirical relationships developed for one alloy do not necessarily generalize to another. Temperature effect, for example, can affect the breakdown potential in an opposite way for two different metal systems (12). A detailed discussion of the significance of this general finding for electrolytically etched Rexillium III is outside the scope of this paper and will be dealt with elsewhere.

The most interesting photometer results were obtained by varying the viewing angle and the viewing aperture. Viewing aperture is known to affect the ability of a reflection photometer to distinguish roughness changes for relatively smooth surfaces (as in surface gloss analysis) (9). The fact that this phenomenon holds for very rough surfaces is interesting. An explanation for this effect may lie in the theory that most biangular reflection intensities are composed of both specular and diffuse components (13). The pure specular component is thought to be calculable by extrapolating a plot of decreasing solid cone angle of irradiation versus reflection intensity to an intercept on the intensity axis (13).

The observation that reflection maximum occur at viewing angles beyond the angle of incidence (Figures 7 and 8) is also not unique to this investigation. A number of authors have reported reflection peaks to occur beyond the angle of incidence for many different diffusing surfaces including metals, ceramics and packed snow (14,15). For some metals this off-specular peak may be three or four times the intensity of the specular peak and its position appears to be a function of surface roughness (16). An

analytical model to explain the off-specular peak includes contributions from three mechanisms: 1) Fresnel reflection from small mirror-like facets; 2) scattering from facets according to Lambert's law; and 3) incomplete illumination of facets due to shadowing by adjacent facets (15).

Results of this study may fall into that observed behavior for diffusing metal surfaces. The etched Rexillium III discs, however, may also be acting as diffraction gratings. A diffraction peak may be evident in Figure 11 that seems to increase in strength at increased etch times. This is being investigated further.

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TABLE 1

CURRENT DENSITY EFFECTS
(180 sec, RT, 10% sulfuric)

Tensile Bond Strengths

Current Density (ma/cm^2)	Bond Strength Means (MPa)	SD
100	5.8	1.1
200	7.6	0.5
300	5.7	1.5
400	5.2	0.4
500	6.1	3.62

TABLE 2

AGING OF ETCH SOLUTION
(300 ma/cm^2, 180 sec, RT)

Test Bridge Etch Periods	Reflection Mean ($microwatts/cm^2$)	SD
0	.129	.007
5X	.131	.004
10X	.128	.011
15X	.129	.007
20X	.129	.004
25X	.134	.004
30X	.134	.004

TABLE 3

METHANOL CONCENTRATION EFFECT
(300 ma/cm^2, 180 sec, RT, 10% sulfuric)

Concentration (volume percent)	Reflection Mean (microwatts/cm^2)	SD
0	.148	.005
5	.150	.005
10	.153	.008
15	.148	.005
20	.143	.005

TABLE 4

VIEWING ANGLE EFFECT
(incidence = 30 degrees, white light)

5 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.535	.025
120	.469	.027
180	.402	
420	.388	.015
840	.391	.010
Control	.187	---

10 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.608	.025
120	.510	.024
180	.434	.028
420	.409	.013
840	.415	.008
Control	.228	---

20 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.733	.021
120	.574	.017
180	.480	.017
420	.435	.005
840	.444	.007
Control	.386	---

TABLE 4 (Continued)

30 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.766	.011
120	.567	.013
180	.484	.013
420	.428	.007
840	.448	.007
Control	.480	---

40 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.766	.005
120	.574	.016
180	.496	.011
420	.439	.004
840	.461	.008
Control	.438	---

50 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.723	.007
120	.552	.018
180	.480	.017
420	.421	.005
840	.445	.007
Control	.415	---

TABLE 4 (Continued)

60 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.687	.012
120	.548	.031
180	.478	.037
420	.426	.005
840	.449	.003
Control	.352	---

70 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.556	.013
120	.469	.028
180	.420	.032
420	.382	.010
840	.396	.005
Control	.290	---

80 Degrees Viewing Angle

Etch Time (seconds)	Reflection Means (microwatts/cm^2)	SD
60	.405	.011
120	.365	.017
180	.338	.019
420	.318	.009
840	.330	.004
Control	.220	---

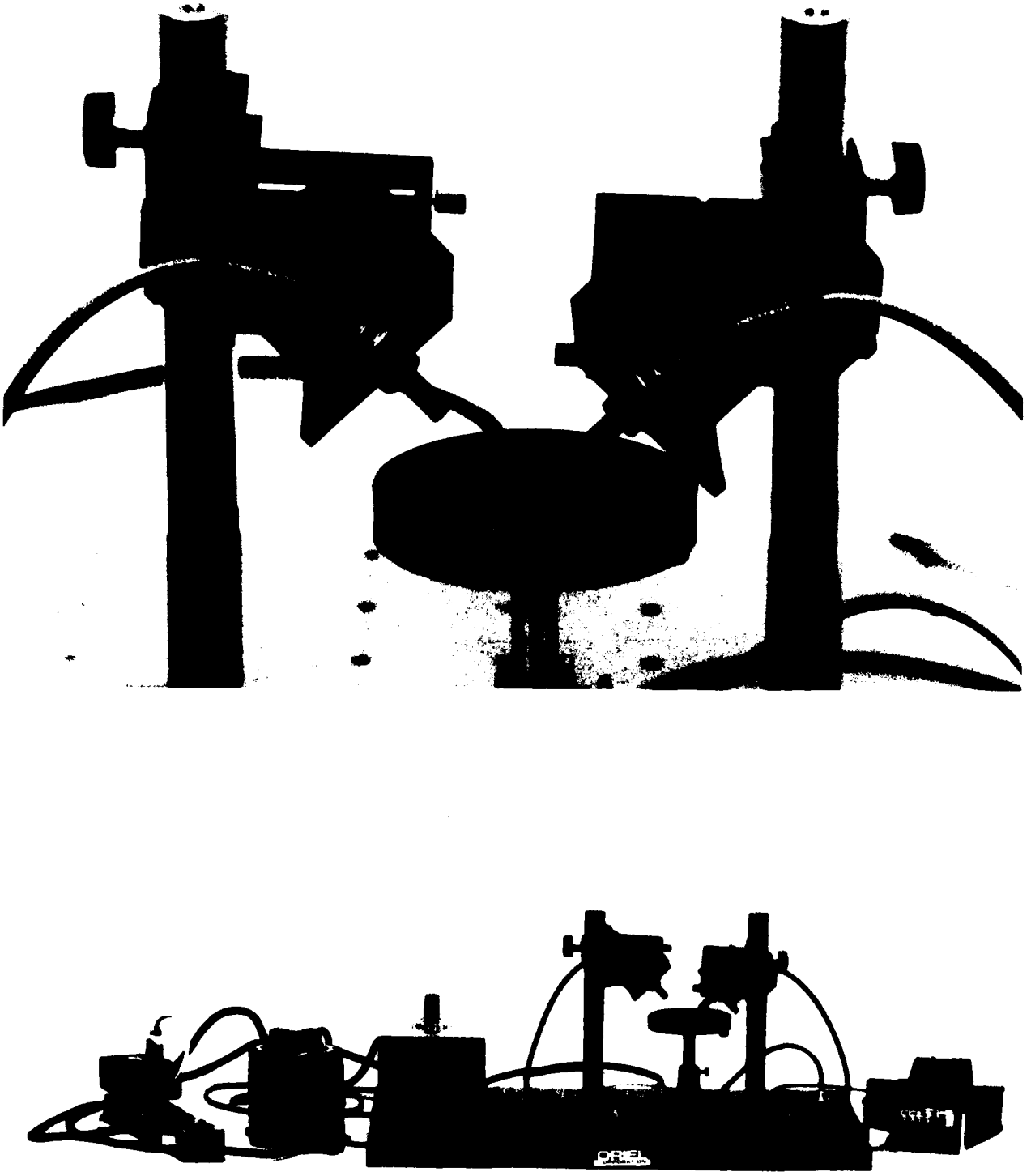


Figure 1. The reflection photometer.

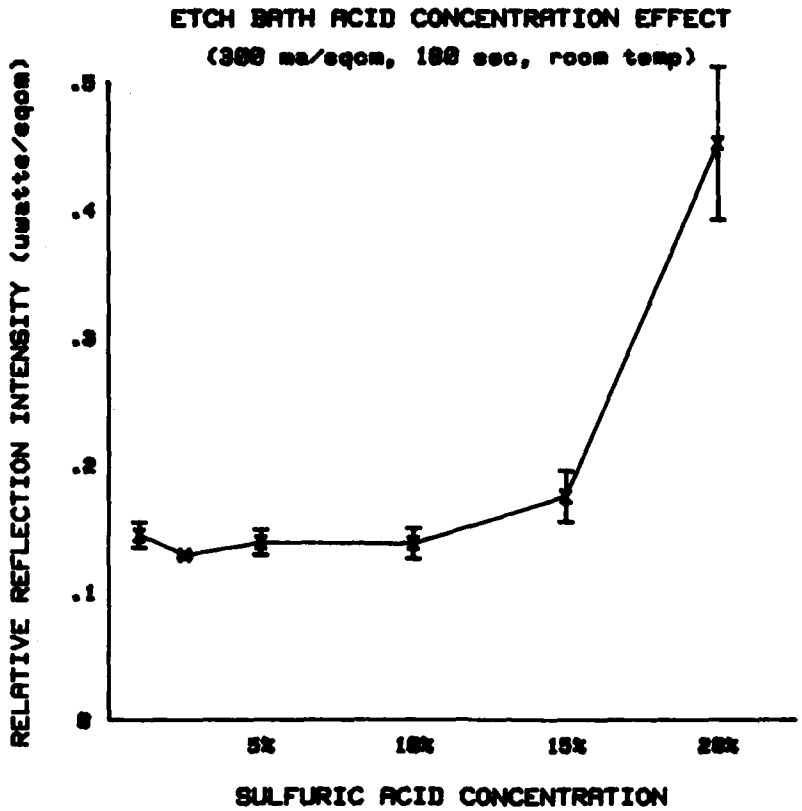
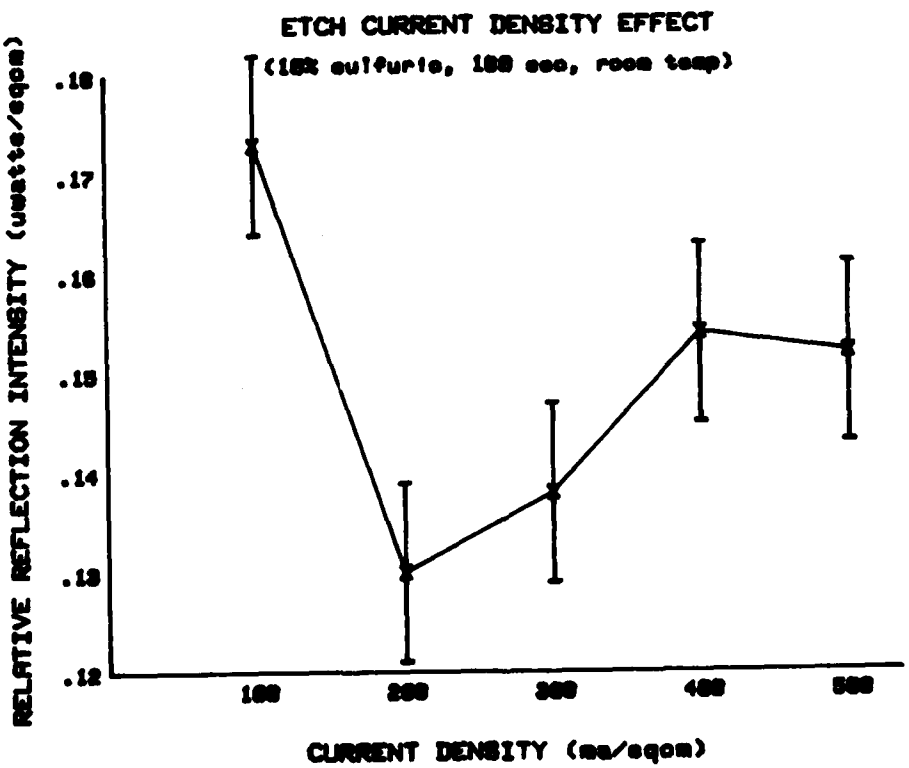


Figure 2.



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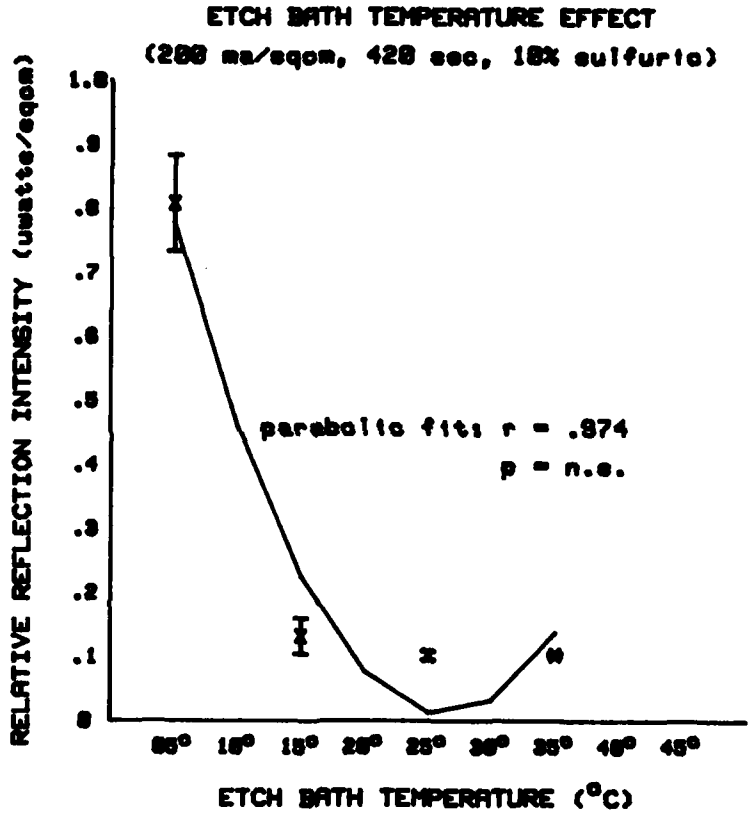
ETCH BATH TEMPERATURE EFFECT
(200 ma/sqcm, 420 sec, 18% sulfuric)
RELATIVE REFLECTION INTENSITY (uwatts/sqcm)
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.9
.8
.7
.6
.5
.4
.3
.2
.1
0
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p = n.s.
10° 15° 20° 25° 30° 35° 40° 45°
ETCH BATH TEMPERATURE (°C)

Figure 4.

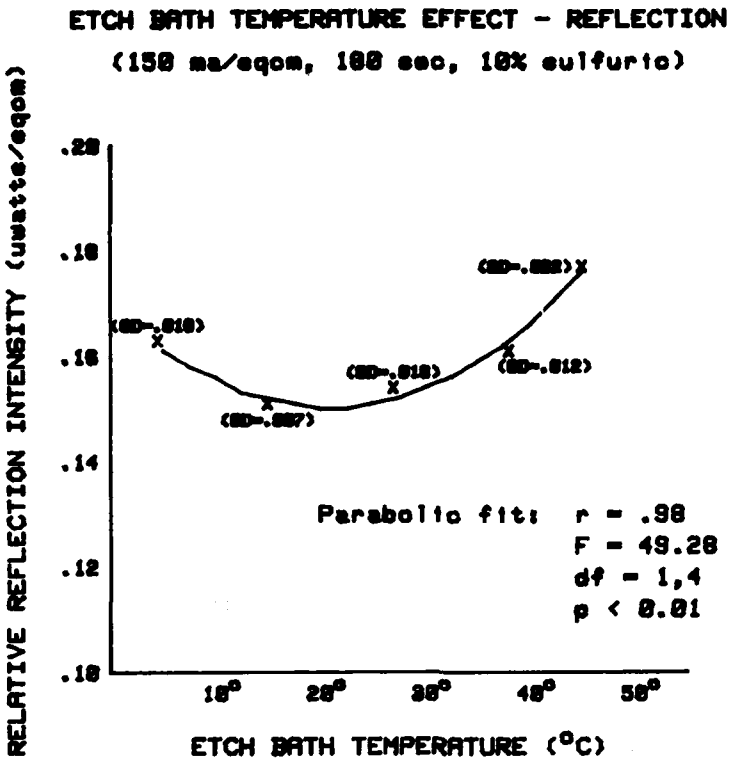


Figure 5.

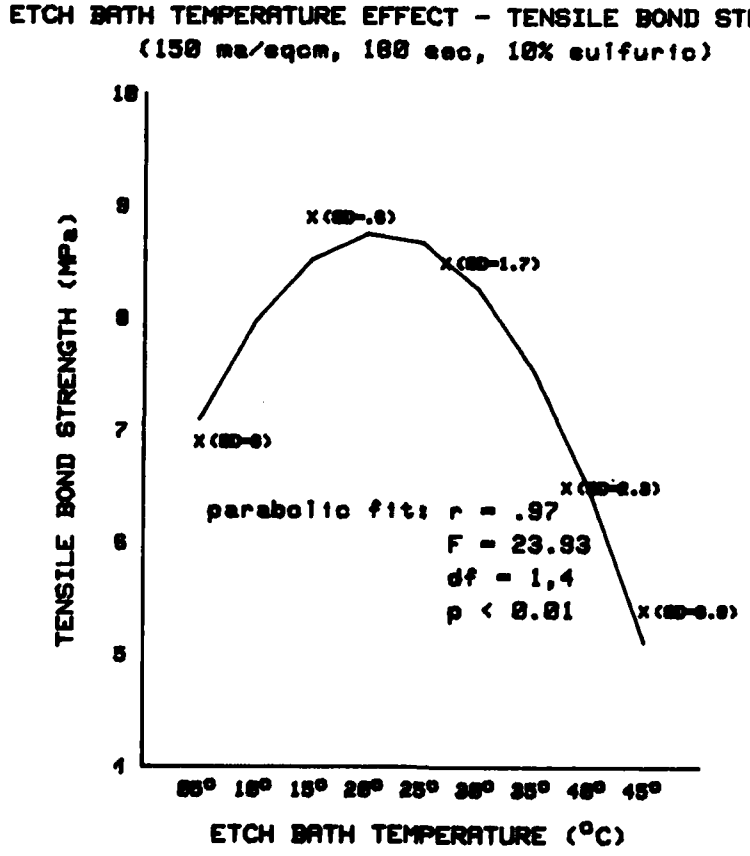


Figure 6.

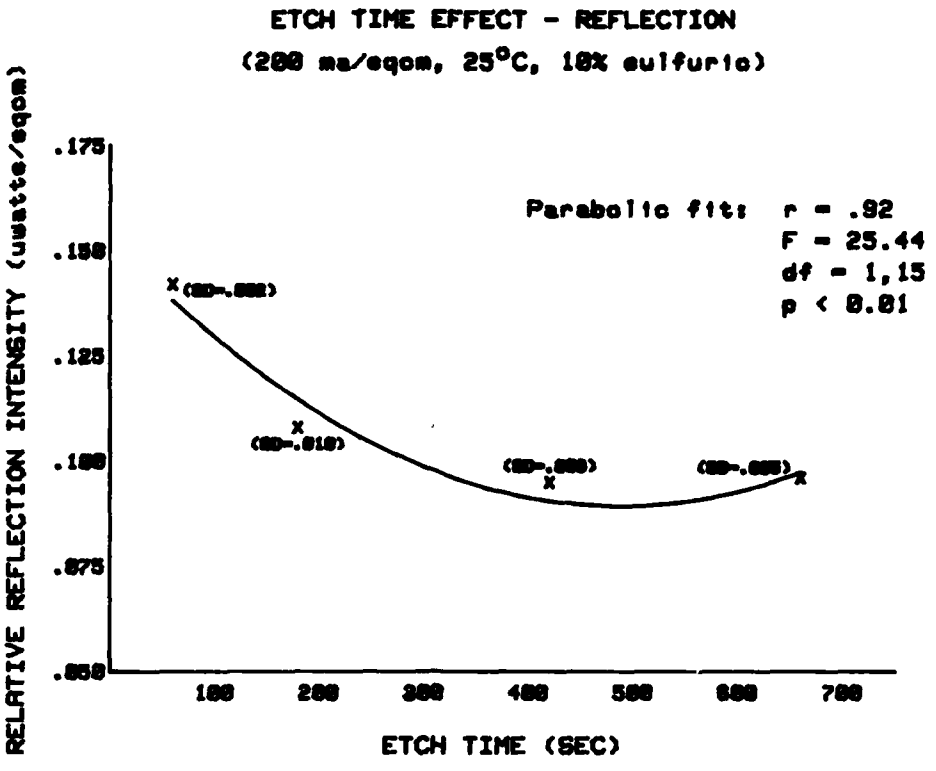
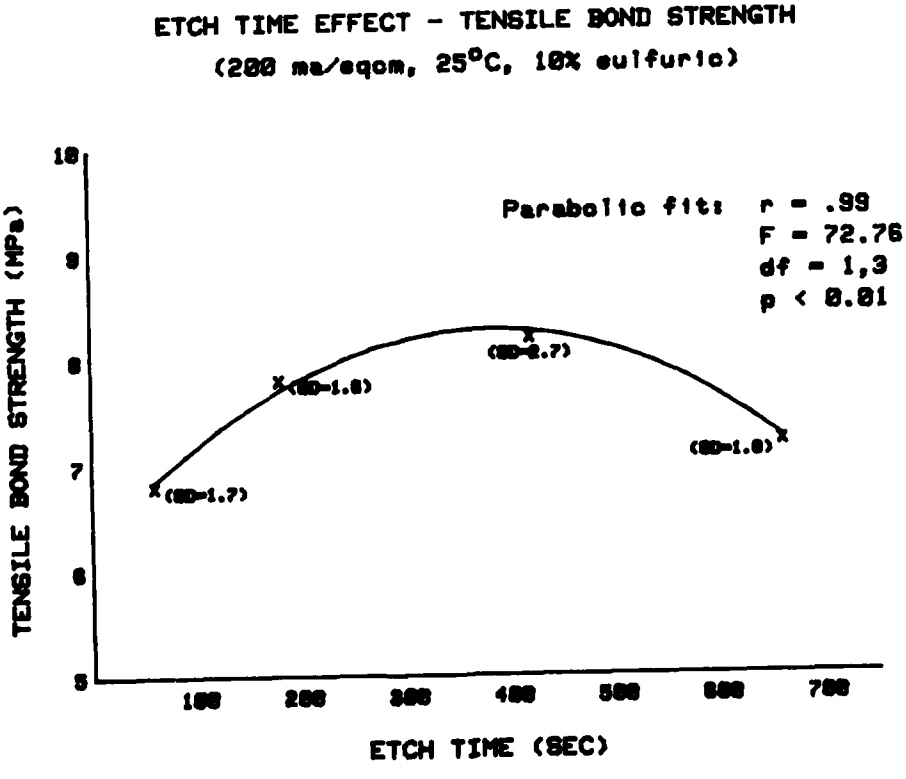


Figure 7.



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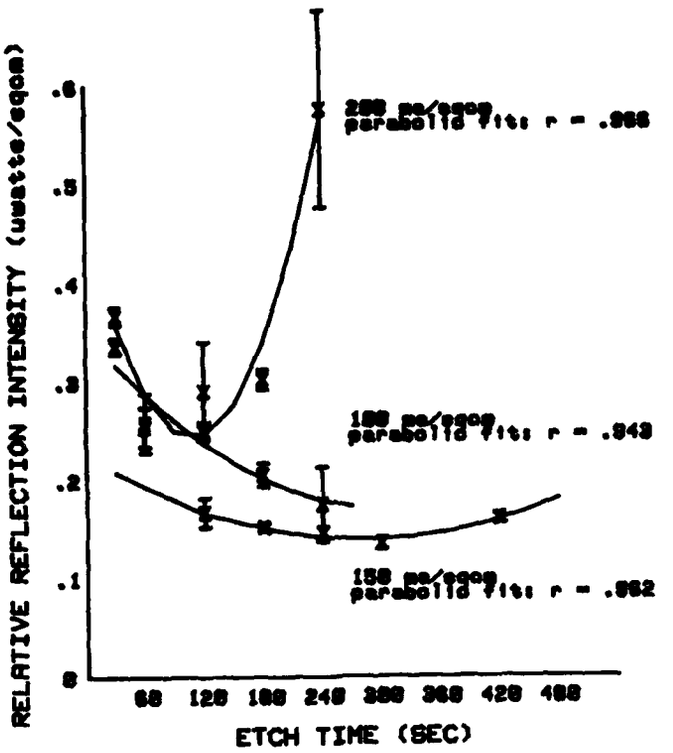

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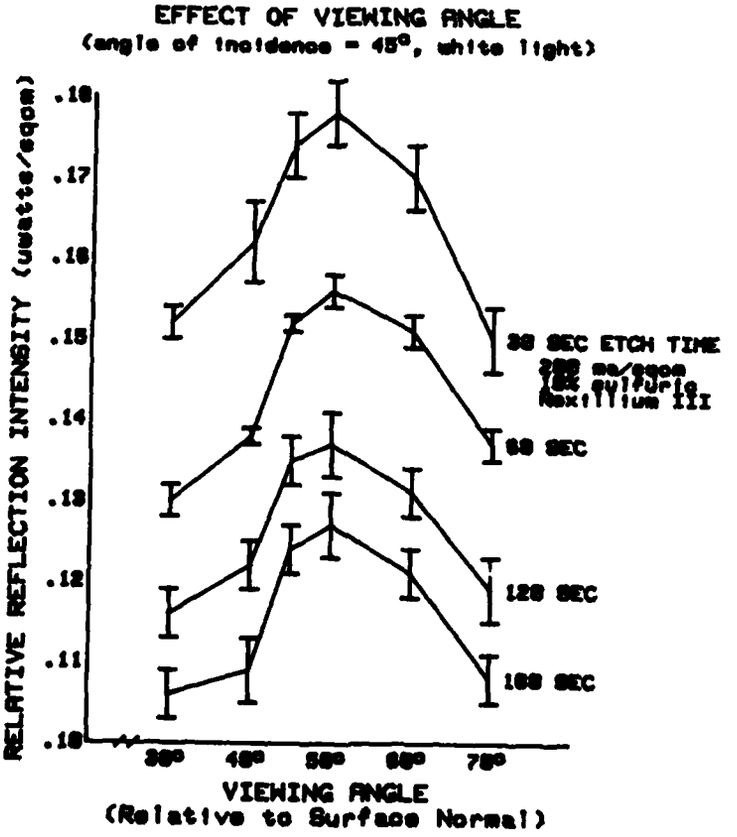

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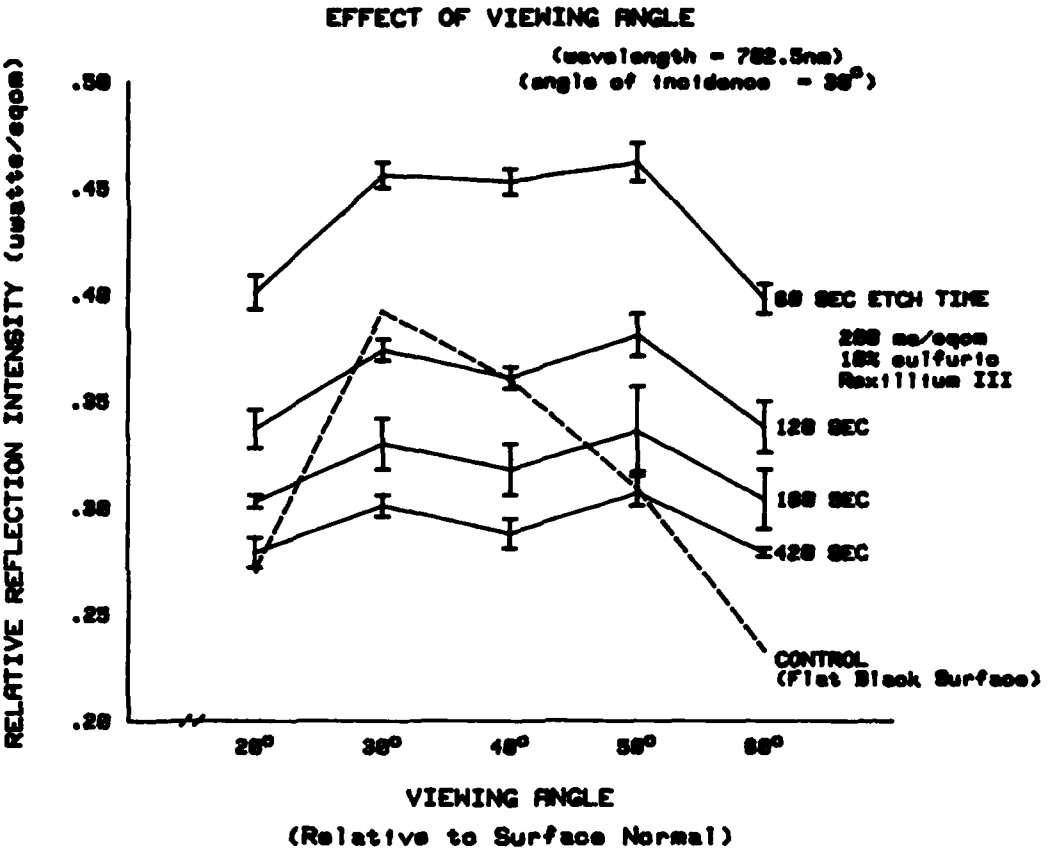

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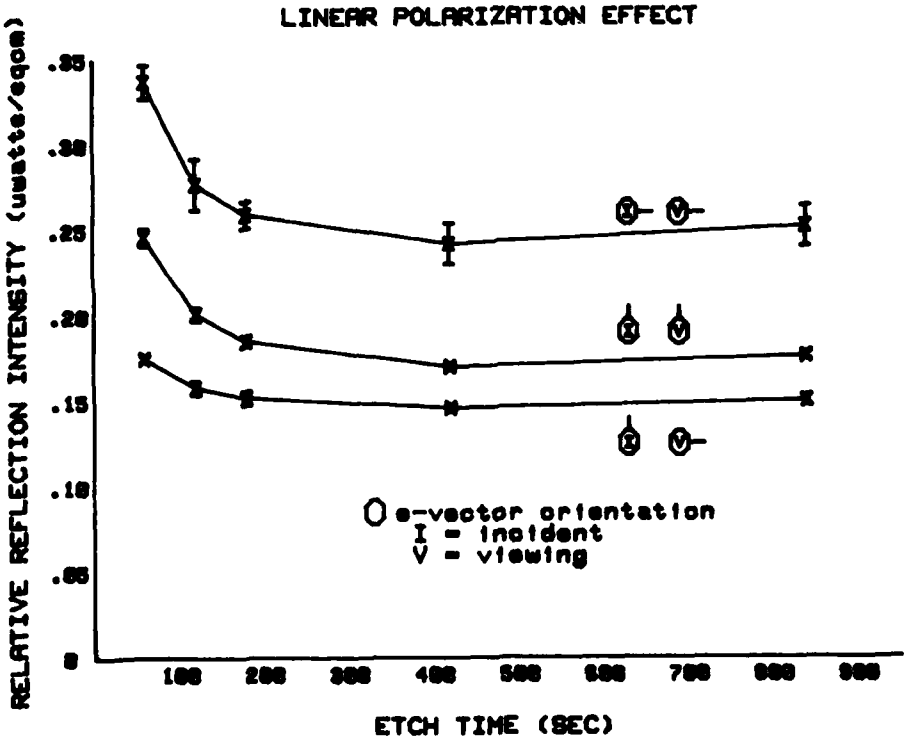

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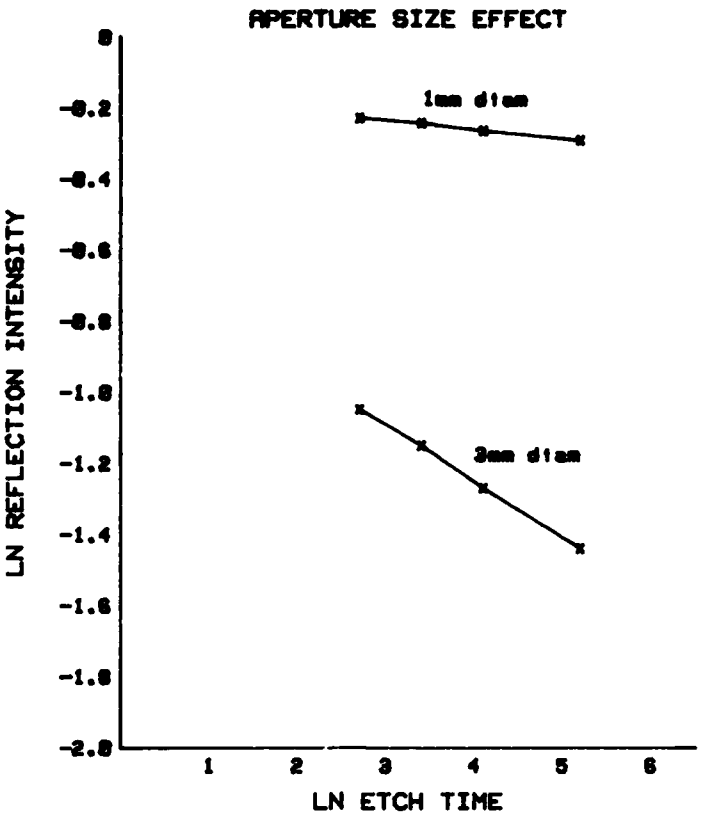

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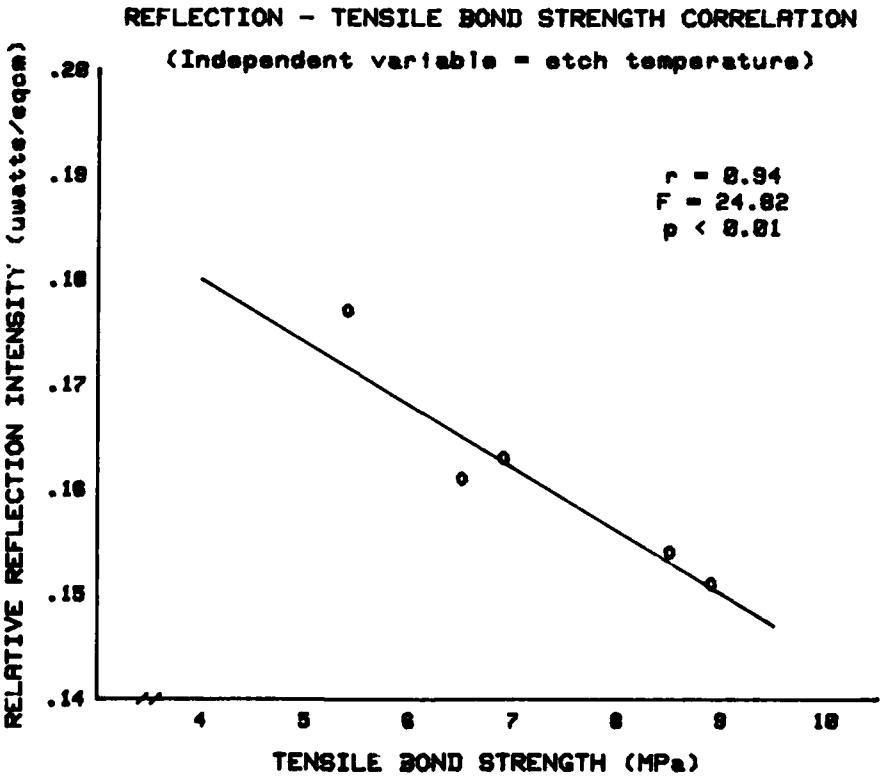

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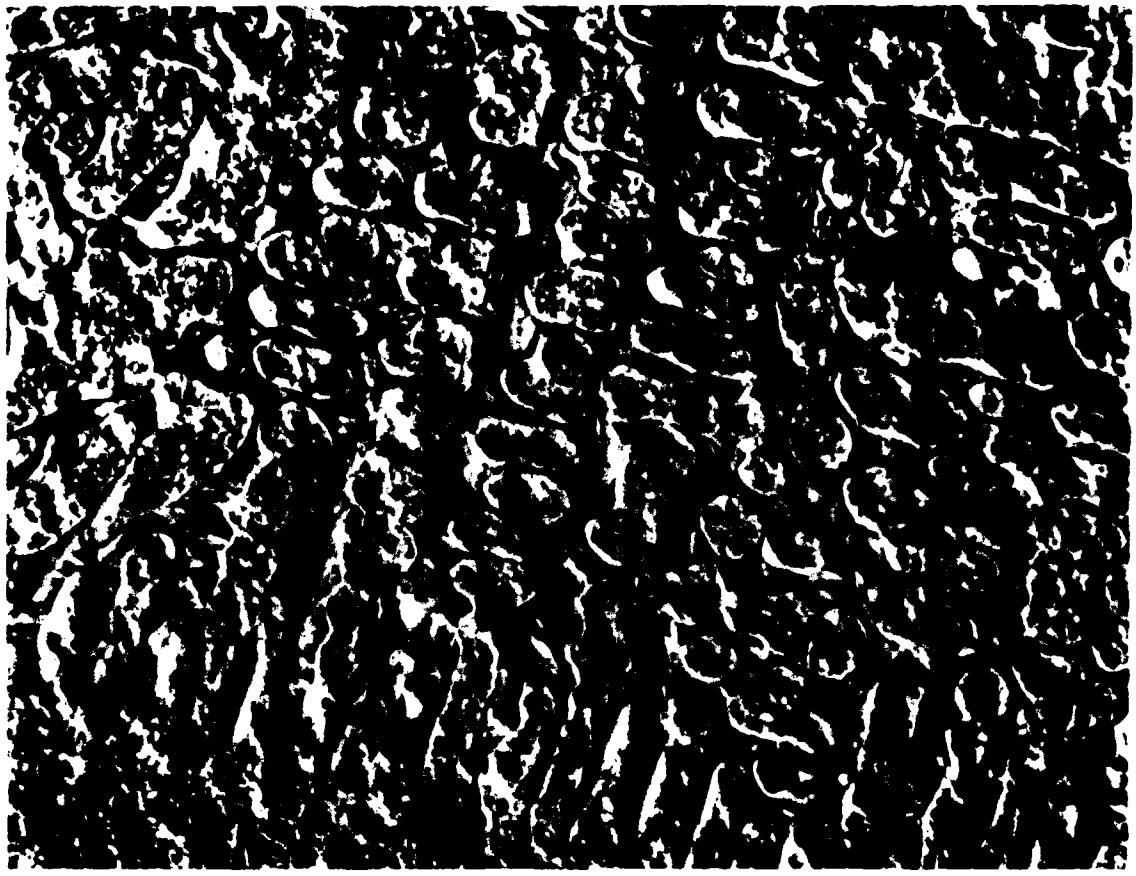

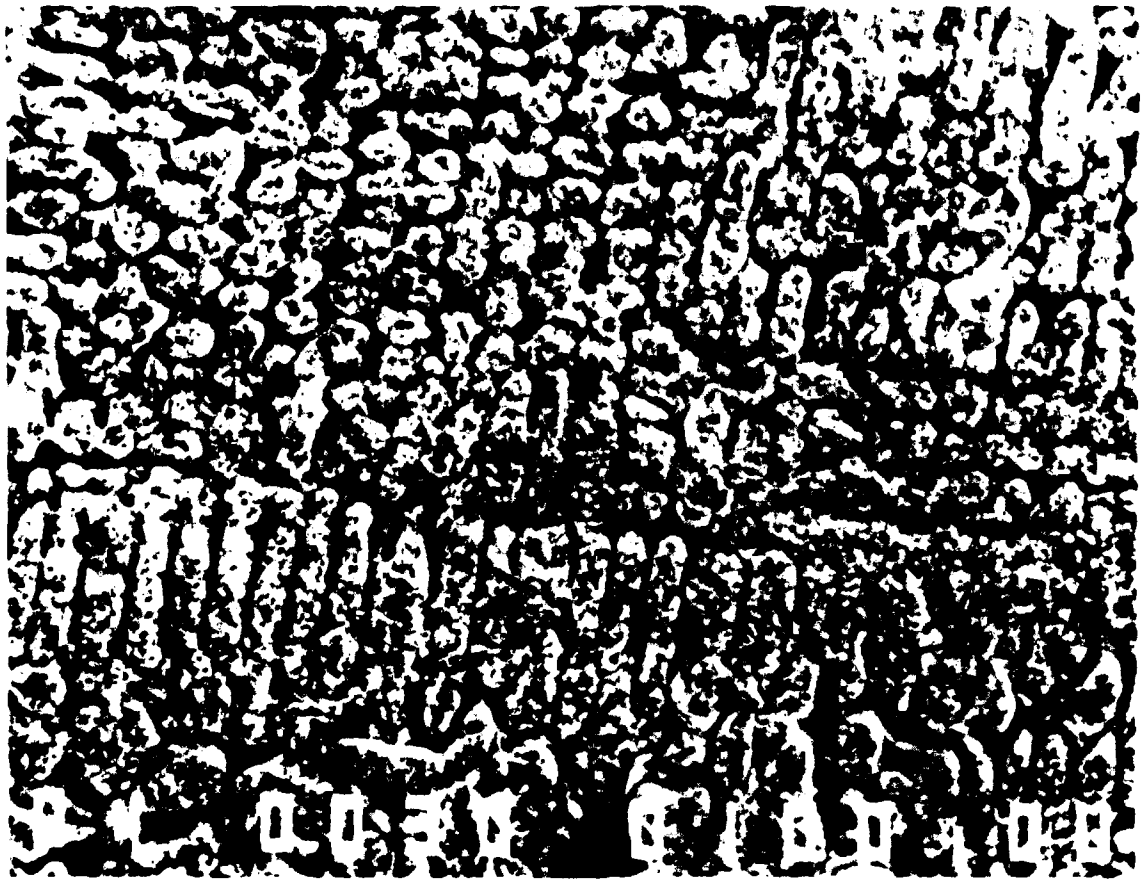

Figure 15. SEM view of etched Rexillium III at 5 and 35 degrees centigrade. Original magnification 200X.

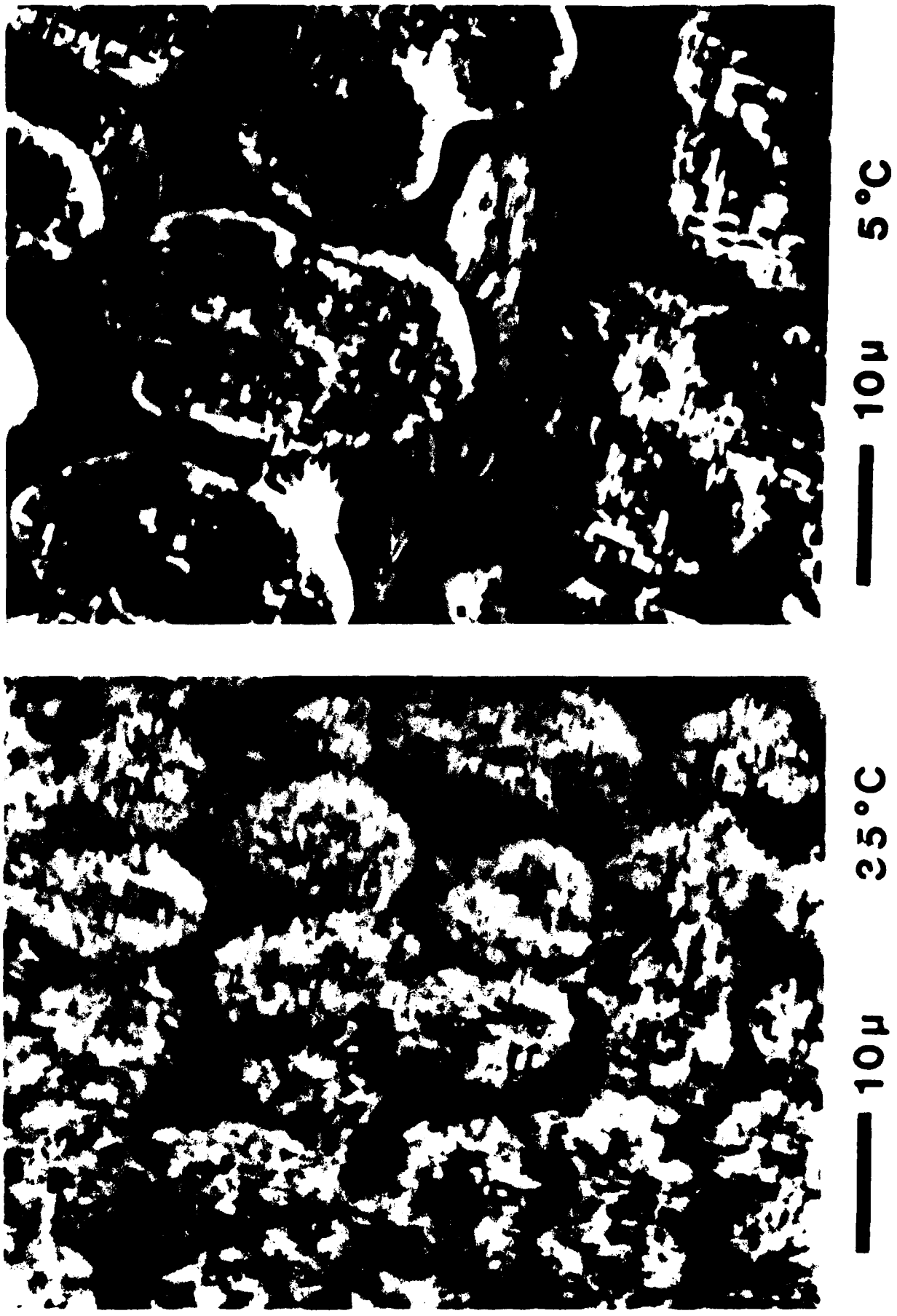

Figure 15 (Cont.). SEM view of etched Rexillium III at 5 and 35 degrees centigrade. Original magnification 1000X.

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18. SUPPLEMENTARY NOTES

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(cont'd on 1473)

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Resin bonding;
Prostheses;
Dental materials.
Casting Alloys;
Electrolytic etching.

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Reflection of light from electrolytically etched casting alloys is empirically related to the incident light angle, viewing and solid cone angles, absorption and the root mean square surface roughness. These factors were evaluated for the further development of a fiber optic photometer to quantitate base metal etching. Dental laboratory etching variables studied include: current density, sulfuric acid and methanol concentrations, temperature, etch time and aging of the etch solution. Resin tensile bond

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strengths and reflection values were correlated by regression analysis.

Peak light intensity occurs above the angle of incidence. Increasing the viewing solid cone angle enhanced reflection sensitivity. Reflection measurements showed current density, acid concentration, etch time and temperature to be critical in controlling the selective removal of interdendritic phase(s). Bond strength values tended to be related to reflection values for some sets of castings. Bond strength values, however, were much lower overall than expected. A significant relationship between clinically meaningful bond strengths and reflection values remains to be determined. Keywords: → (illegible)

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